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Determination of Trace  
Organics in Well Waters Collected  
Around the Llangollon Landfill

ORIGINAL *Report Full  
of Errors*

RECEIVED

by

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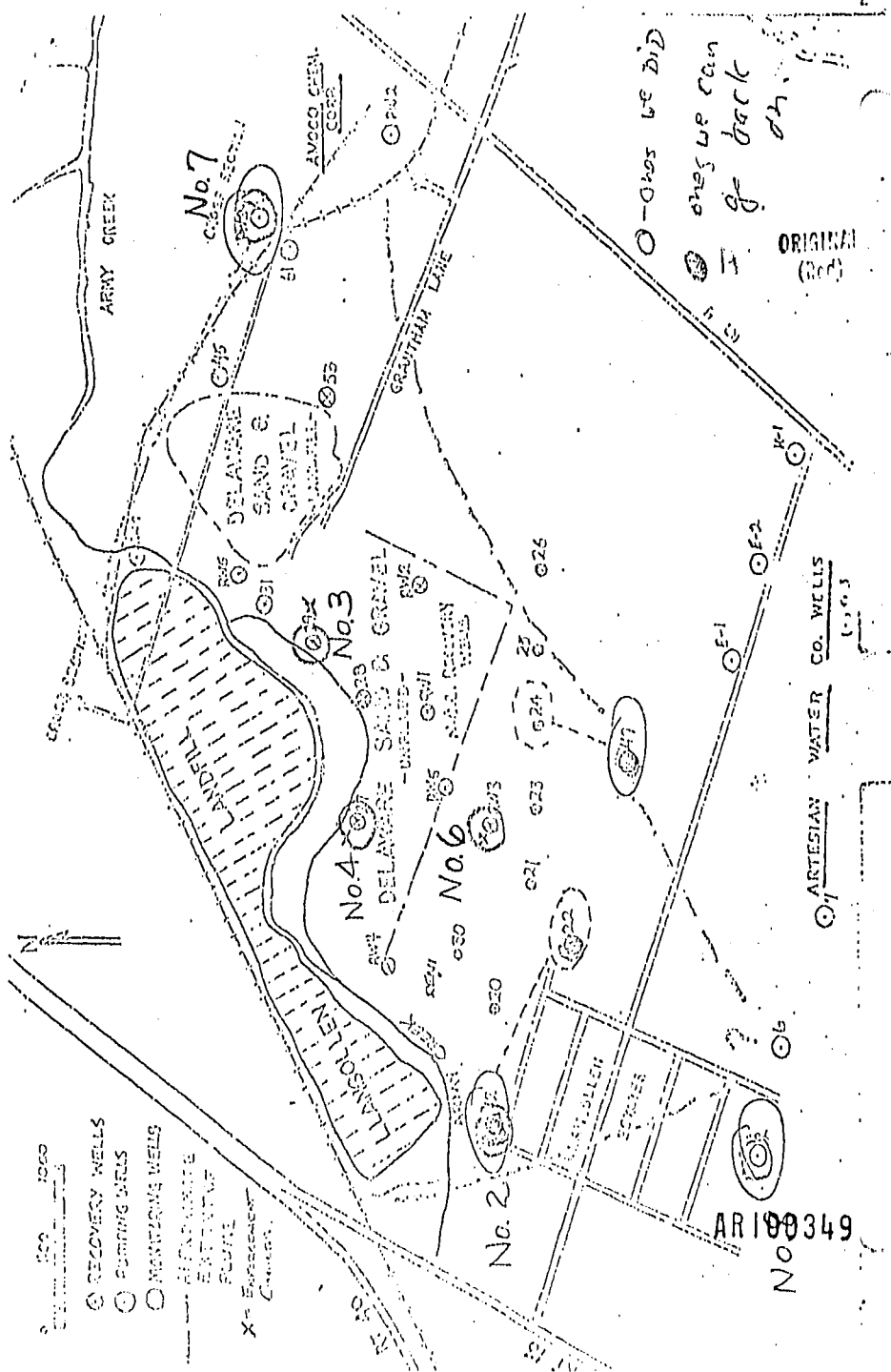
SAMPLE COLLECTION

Dr. Tom Munson of the Annapolis Field Office, Annapolis Science Center, Region III, Environmental Protection Agency sent us on February 24, 1977 six groundwater samples collected from different sites around the Llangollon Landfill for analysis of trace organics. Each sample was shipped in a one-gallon glass container and in a 4-oz. vial. Due to the unavailability of an oven to muffle the one-gallon solvent containers, all of them were cleaned by rinsing with n-hexane solvent. Figure 1 gives the specific locations from which these samples were collected. They are marked by sample numbers 2, 3, 4, 6, 7, and 8 (Figure 1).

SAMPLE PREPARATION AND ANALYSIS

Each sample was subject to volatile organic analysis (VOA) and determination of organics in the acid and the base extracts. Figure 2 gives the procedures for stripping of volatile organic compounds. Figure 3 shows a schematic of the elution system for the

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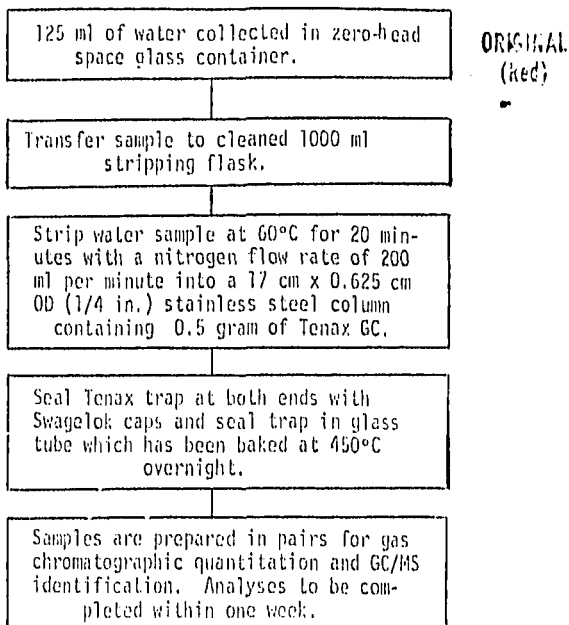
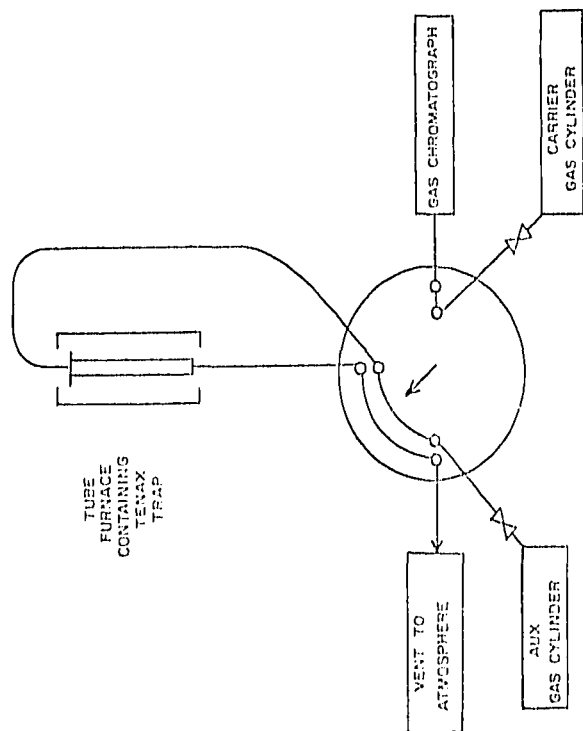


Figure 2 Protocol for Stripping of Volatile Organic Compounds in Aqueous Samples.

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Figure 3 Schematic Diagram of Volatile Organic Elution System



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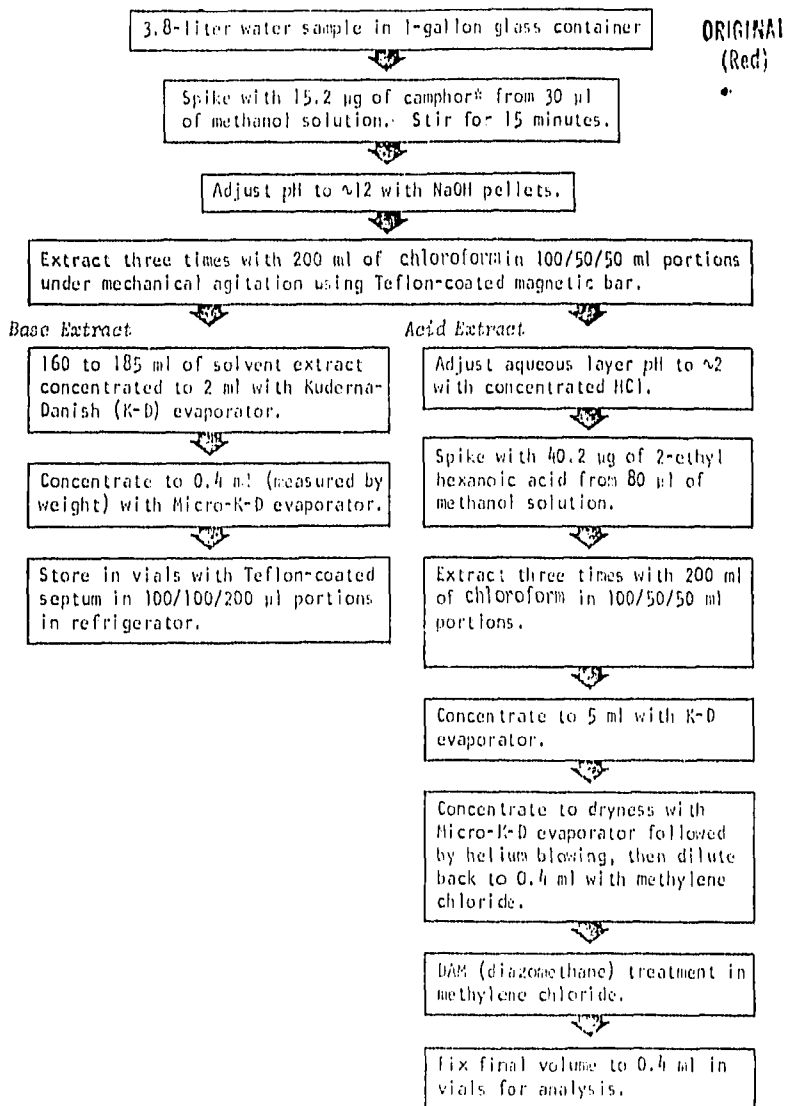
stripped volatiles from the Tenax-GC traps. The valve, supplied by Valco, is a two-position, six-port valve which can be heated to 200°C. When installed, both the Tenax-GC trap and the valve can be heated to 250°C while the valve, transfer lines and GC inlet are held at 200°C. The carrier flow to the GC column is not interrupted and the column is never exposed to the atmosphere. Also, traps can be swept with carrier gas before connecting them to the GC column, again eliminating exposure of the column to the atmosphere and in the GC/MS runs eliminating water vapor. The zero-dead volume construction of the valve virtually eliminates losses of sample and cross-contamination between samples.

The volatile organics were analyzed on a 365 cm (12-foot) by 2-mm-ID glass column packed with 0.2% Carbowax 1500 on 60/80 mesh Carbowax C (Supelco, Inc., Bellefonte, PA). The trapped compounds were desorbed from the Tenax-GC column trap at a peak temperature of 250°C for six minutes with a helium flow of 40 ml/min. The glass GC column was held at ambient temperature during this period. The oven temperature was then programmed to increase at 8°C/min to 200°C and held there for the remainder of the run. Mass spectra were acquired for m/e 33 to m/e 350 at a scan rate of 2.3 seconds per mass decade and at an ionization potential of 70 eV on a Varian-MAT 311A GC/MS. Data were acquired on a Varian-MAT SS100 data system and stored on a magnetic tape or disk cartridge for later analysis with ADP-Cyphenetries Mass Spectral Search System (MSSS).

The less volatile organic compounds present in the aqueous samples were recovered by a series of extractions as outlined in Figure 4. The extractions shown allow the separation and identification of a large number of compounds. Samples of 3.0 liters were spiked with camphor as the internal standard. The pH was adjusted to 12, and then the sample was extracted with spectra grade chloroform (Burdick and Jackson Lab, Muskegon, MI).

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\*Low recovery (~70%) due to enolization at high pH. The sensitivity for camphor on OV-17 columns and the FID is higher than for naphthalene.

Figure 4 Procedure for base and acid extraction of the less volatile organics.

The solvent was thus evaporated to 2 ml in a Kuderna-Danish evaporator (Kontes, Vineland, NJ), and, subsequently, to 0.4 ml in a micro-Kuderna-Danish evaporator. This sample was subject to quantitation by gas chromatography and identification by GC/MS. The aqueous sample was further acidified to pH 2 and after the addition of a second internal standard (2-ethyl hexanoic acid) it was extracted with chloroform. It was evaporated to 5 ml in a Kuderna-Danish evaporator. Methylation of the concentrated acid extract was carried out in a hood using purified diazomethane. Prior to methylation chloroform was replaced by methylene chloride. The final volume was adjusted to 0.4 ml and the sample was examined by the GC and GC/MS procedures to separate, quantitate, and identify the various organic species present. The solvent extracts were analyzed on a 366-cm (12-foot) by 2-mm-ID glass column packed with 3% OV-17 on Supelcoport 80/100 mesh (Supelco, Inc., Bellefonte, PA). One microliter of extract was injected on column. The column temperature was held at 50°C for 6 minutes and then programmed at 10°C/min to 300°C, where it was held for the remainder of the analysis. The specific column and GC conditions employed here, however, do not provide good separation of low MW volatile fatty acids, i.e., C<sub>1</sub>-C<sub>6</sub> monocarboxylic acids, since the C<sub>8</sub> fatty acid internal standard appears in a very early stage of the GC run. Other columns and GC conditions should be used for determining these low MW volatile fatty acids. MS scans were made from m/e 33 to m/e 600 with the same conditions as in the analysis of VOA.

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#### RESULTS AND DISCUSSION

Results of organic analysis of these samples are given in Tables 1 to 18. Table 19 summarizes the overall rating of the level of contamination

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of each sample based on the number of organic compounds present in concentrations of above 1 µg/l (ppb) in all three fractions, i.e., VOA, acid and base extracts. It is seen from Table 19 that water sample No. 8 from the Artisian well appears to be the cleanest. This is followed by sample No. 2 from the Monitoring Well #42. Sample No. 3 and No. 4 represent the most contaminated water samples among all six analyzed. They are followed by sample No. 7 and No. 6 which are somewhat less contaminated than sample No. 3 and No. 4 and are more contaminated than sample No. 8 and No. 2. The fact that the sample No. 2 is much cleaner than the corresponding samples (No. 3 and No. 4) collected from well located immediately around the landfill, indicates that either the groundwater moves in the south-eastward direction as shown on the map (Figure 1) or the landfill around the Monitoring Well #42 (sample No. 2) is more stabilized or is located somewhat higher above the water table than that where the other two wells are located.

Since only one bottle of 4-oz. vial, instead of 3 as required for VOA, was received for each sample, they were used mainly for quantitation purposes. The VOA samples prepared for GC/MS identification were actually taken from the one-gallon glass containers previously used for containing various solvents, such as hexane, methylene chloride, chloroform, and acetone as indicated by the labels on these bottles. Because the high background contamination levels exerted by these solvents greatly impaired the GC/MS analysis, only 5 ml of the water samples from the one-gallon glass containers were used for VOA analysis. This reduces greatly the sensitivity of the VOA method employed in this study. Also, the **CART-00355** e.g., n-hexane, methyl pentane, and hexane-1, have been found ubiquitously in all VOA samples, suggesting contamination by the n-hexane used for rinsing all these one-gallon glass containers. The presence of high



concentrations of methylene chloride, chloroform and acetone in VOA of these samples also suggests carrying-over from the glass containers previously used for containing these solvents. Since the detection limit of the specific VOA method employed in this study is as low as 0.1 ppb, they are highly susceptible to contamination by other volatile organic solvents. Even a small quantity of solvent vapors normally present in the laboratory environment would tend to contaminate the VOA analysis.

The detection of a number of phthalates, e.g., dibutyl, di-(2 ethyl hexyl), diethyl, etc., in both the acid and the base extracts may be a result of contamination from the plastic materials (e.g., plasticizers) used in the lid and the liner of the one-gallon glass solvent bottle, or that used in the well casing, pump parts, and transfer tubings. In future samplings a piece of Teflon liner should be placed between the lid and the container and efforts to avoid using any plastic parts, except Teflon, in sample collection should be made.

The fact that a large number of the unidentifiable peaks present in the acid and the base extracts of samples No. 3 and No. 4 is rather discouraging. This is actually limited by the mass spectra data available in the Mass Spectra Search System (MSSS) employed in this study. Although the MSSS is considered to be one of the most comprehensive library (35,000 chemical compounds) for MS search, the last time the MSSS was revised was about two years ago. A large number of mass spectra data have since been developed by the US EPA Laboratories at Athens and Cincinnati which have not yet been incorporated into the current MSSS. Nevertheless, all of the bar charts and mass lists for these unknowns are available. **ARI-00956** identification of these compounds. Another approach is to use glass capillary columns to better resolve these peaks so that they can be identified more positively. We are currently in the process of developing

capillary glass high-resolution columns which can be used for separating a few of the highly contaminated water samples. The SCOT and the WCOT high resolution glass capillary columns will be studied.

#### RECOMMENDATIONS

In order to eliminate any confusion in the future, the water samples shall be collected according to the following procedures.

1. The specific sampling sites shall be selected in consultation with Dr. F. B. DeWalle
2. A 1-gallon Pyrex glass container shall be used to store the water sample in which the organics in the acid and the base extracts will be determined. This container shall be muffled (450°C) overnight prior to use. In the event when a muffled furnace is not available, a new 1-gallon Pyrex container shall be used. In no circumstance shall the organic solvent bottles be used for sample collection. A small piece of Teflon liner shall be placed between the lid and the container to minimize contamination by the lid.
3. Three (3) muffled (450°C, overnight) 4-oz serum bottles with zero-dead volume and Teflon faced septum shall be used to store the water sample in which the volatile organics will be determined
4. Prior to sample collection, all containers and vials are to be rinsed several times with the water sample to be collected. The use of any plastic materials, with the exception of Teflon shall be avoided in sample collection. No preservatives shall be added. The sample shall be refrigerated or iced down as soon as it is collected
5. All samples are to be shipped air-freight within one day after collection to the attention of

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3217 Civil Engr. Bldg.  
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Urbana, IL 61801

Upon delivery of the samples for shipping, it should be followed up with a telephone call (217-333-6962) (AR 00357) Dr. Chian of the approximate time of sample arrival at the Urbana-Champaign Airport so the proper arrangement can be made to pick up the samples.

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Table 1

Llangollen Landfill Sample No. 2, VOA

Monitoring Well #42  
(Total number of Peaks > 1 PPb 20)

Relative Retention Time*	Compound	Approximate Level (PPb)	(Hexane bottle)
0.59	Acetone	1	
0.95	Tetrahydrofuran	6	
1.00	Chloroform	32	from P 11
1.01	Dichloroethene	< 1	
1.15	Dichloroethane	< 1	
1.17	Methyl ethyl ketone	< 1	
1.33	Hexene-1	32	(mostly 20 PPb from hexane bottle)
1.40	Trimethyl Silol	1	
1.52	Methyl pentane	32	
1.57	Benzene	5 ?	(w/ 100) FROM P 11
1.58	Trichloroethene	5	
1.71	n-Hexane	40	
1.94	Hexanone (Pinacolone)	1.5	
2.05	3-Hexanone	1	
2.10	2-Hexanone	< 1	
2.17	Toluene	1	
2.21	n-Hexanol	1	

\*Relative to Chloroform

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Table 2  
Llangollon Landfill Sample No. 2, Acid Extract  
Monitoring Well #42  
(Total Number of Peaks > 1 PPb 7)

Relative Retention Time*	Compound	Approximate Level (PPb)
0.42	2-Ethyl hexanoic acid (Is) <sup>at 0.42</sup>	--
0.69	Acenaphthene (or Diphenyl)	5
0.76	C10 Terpeneol	10
0.92	Methyl palmitate	1.5
1.00	* Dibutyl phthalate + Methyl Stearate	3
1.01	Bleed	--
1.05	Bleed	--
1.22	* di-(2-ethyl hexyl) phthalate	3
1.28	C15 Terpeneol	1

\*Relative to methyl stearate

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Table 3  
Llangollon Landfill Sample No. 2, Base Extract  
Monitor Well #42  
(Total Number of Peaks > 1 PPb 7)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
0.13	Diethoxyethane	3
0.35	Dichlorobutane	21
0.37	Unknown	< 1
0.51	Camphor (IS)	-
0.76	C <sub>10</sub> - Terpineol	1
0.83	Diethyl phthalate	3
1.00	Dibutyl Phthalate	2
1.15	Bleed	1
1.22	C <sub>15</sub> terpineol	1

\*Relative to methyl palmitate

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Table 4  
 Llangollon Landfill Sample No. 3, VOA  
 Monitoring Well #42  
 (Total Number of Peaks > 1 PPb 29)

Relative Residence Time*	Compound	Approximate Level (PPb)
0.54	• Acetone	good 1180 (methylene chloride)
0.94	Diethyl ether	63
1.00	• Chloroform	881 good, low to mid
1.26	Hexene - 1	251
1.32	Methyl-Tetrahydrofuran	10
1.36	Trimethyl Silol	5
1.44	? Methyl Pentane	13
1.49	Benzene	2
1.63	• Hexane	923
1.97	3-Hexanone	130
2.02	2-Hexanone	98
2.10	Toluene	56
2.13	n-Hexanol	61
2.24	Chlorobenzene	4

\*Relative to chloroform

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Table 5

Llangollon Landfill Sample No. 3, Acid Extract

Recovery Well #29

(Total Number of Peaks &gt; 1 PPb 41)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
0.35	Unidentified	28
0.41	2-Ethyl hexanoic acid <sup>***</sup>	--
0.45	Unidentified	< 1
0.47	"	< 1
0.56	"	< 1
0.69	"	5
0.71	Bleed	--
0.73	Unidentified	1
0.79	C <sub>4</sub> substituted Naphthalene	1
0.81	Unidentified	1
0.82	"	10
0.86	"	16
0.89	"	8
0.92	Methyl palmitate	9
0.93	Unidentified	8
0.95	"	19
1.0	Dibutyl phthalate + Methyl Stearate	24
1.03	Unidentified	8
1.06	C <sub>15</sub> H <sub>26</sub> O(C <sub>15</sub> Camphor Type)	19
1.12	Unidentified	8
1.15	Bleed	--
1.17	Butyl phthalyl butyl glycolate	11
1.90	Unidentified	14
1.20	Methyl abietate	10
1.22	di(2-ethyl hexyl) phthalate	19
1.24	Unidentified	10
1.25	"	11
1.26	"	22

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Table 5 (continued).

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
1.26	C <sub>15</sub> terpineol	21
1.29	Unidentified	21
1.31	C <sub>6</sub> H <sub>12</sub> , Polyaromatic hydro- carbons	30
1.33	Unidentified	21
1.36	"	36
1.38	"	27
1.40	"	91

\*Relative to Methyl Stearate

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Table 6  
 Llangollen Landfill Sample No. 3, Base Extract  
 Recovery Well #29  
 (Total Number of Peaks > 1 PPb 42)

Relative Retention Time*	Compound	Approximate Level (PPb)
0.11	Diethoxyethane	1360
0.15	Unidentified	1
0.33	Dichlorobutane	10
0.34	Unidentified	14
0.40	Dichlorobenzene	13
0.46	Unidentified	18
0.48	"	15
0.50	Camphor (1S)	--
0.55	Unidentified	3
0.57	"	1
0.82	"	10
0.89	"	20
0.93	"	12
0.95	"	10
1.0	Diethylphthalate	2
1.04	Bleed	1
1.07	Unidentified	45
1.11	Bleed	--
1.15	Bleed	--
1.16	Butyl Phthalyl butyl glycolate	2
1.22	di-(2-ethyl hexyl) phthalate	1
1.36	Bleed	--

\*Relative to Methyl Stearate

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Table 7  
Llangollon Landfill Sample No. 4, VOA  
Recovery Well #27  
(Total number of Peaks  
> PPb 27)

Relative Retention Time *	Compound	Approximate Level (PPb) <i>(methanol chloride bottle)</i>
0.42	Methylene Chloride	> 2500 - could be good
0.53	Acetone	1100
1.00	Chloroform	5
1.24	Hexene-1	310
1.34	Trimethylsilol	4
1.41	Methyl Pentane	28
1.45	Benzene	3
1.60	N-Hexane	975
1.81	Hexanone (Pinacolone)	4
1.92	3-Hexanone	5
1.96	2-Hexanone	6
2.04	Toluene	1
2.07	n-Hexanol	39
2.32	bis (2-chloroethyl) ether	4
2.35	Xylene (or Ethyl Benzene)	1

\*Relative to Chloroform

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Table 8  
 Llangollon Landfill Sample No. 4, Acid Extract  
 Recovery Well #27  
 (Total Number of Peaks > 1 PPb 22)

Relative Retention Time*	Compound	Approximate Level (PPb)
0.33	Dichlorobutane	21
0.40	2-Ethyl hexanoic acid (15) <i>advised</i>	--
0.43	Methyl pentanoate	< 1
0.46	Unidentified	1
0.56	"	1
0.67	"	2
0.69	"	165
0.76	C <sub>10</sub> Terpineol	3
0.82	Bleed	--
0.87	Unidentified	1
0.91	Methyl palmitate	2
1.00	Dibutylphthalate + Methyl Stearate	2
1.02	Bleed	--
1.05	Bleed	--
1.20	Phthalate (unidentified cpd)	3
1.22	Di (2-ethyl hexyl) phthalate	4
1.25	Bleed	--
1.28	C <sub>15</sub> Terpeneol	21
1.31	Bleed	--
1.36	Unidentified	26
1.41	"	169

\*Relative to Methyl stearate

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Table 9  
Llanqollon Landfill Sample No. 4, Base Extract  
Recovery Well #27  
(Total Number of Peaks > 1 PPb 40)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
0.20	Diethoxyethane	25
0.30	Dichlorobutane	20
0.32	Unknown	10
0.49	Camphor (1S)	--
0.63	Unknown	49
0.85	Bleed	28
0.93	Unknown	19
0.98	Bleed	10
1.0	✓ Dibutylphthalate	11
1.03	Bleed	15
1.09	Bleed	10
1.13	Bleed	16

\*Relative to Methyl Palmitate

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Table 10  
Llangollon Landfill Sample No. G, VOA  
Recovery Well #3  
(Total Number of Peaks > 1 PPb 28)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u> <i>(acetone bottle)</i>
0.45	Methylene Chloride	>2500 <i>3000</i>
0.56	Acetone	> 10 <i>(bottle from bottle)</i>
1.00	Chloroform	2 <i>3000</i>
1.06	Dichloroethane	700
1.25	Hexene-1	420
1.47	Benzene	82
1.62	n-Hexane	550

\*Relative to Chloroform

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Table 11  
Llangollen Landfill Sample No. 6, Acid Extract  
Recovery Well #3  
(Total Number of Peaks > 1 PPb 5)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
0.43	2-Ethyl hexanoic Acid (IS) <sup>1</sup>	--
0.70	Unidentified	14
1.00	Dibutyl Phthalate + Methyl Stearate	1
1.21	Di-(2-ethyl hexyl)phthalate	5

\*Relative to Methyl Stearate

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Table 12  
Llangollon Landfill Sample No. 6, Base Extract  
Recovery Well #3  
(Total Number of Peaks > 1 PPb 9)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
0.17	Diethoxyethane	7
0.34	Dichlorobutane	4
0.35	Unknown	1
0.51	Camphor (15)	-
1.0	( Dibutyl phthalate	1
1.15	Bleed	3
1.16	Butylphthalyl butyl glycolate	2
1.22	di (2-ethyl hexyl)phthalate	100

\*Relative to Methyl palmitate

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Table 13  
Llangollon Landfill Sample No. 7, VOA  
Amoco PW 3 Well  
(Total Number of Peaks 1 PPb 24)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
0.44	Methylene chloride	> 2500
0.58	Acetone	1700
1.00	Chloroform	6
1.22	Hexene-1	890
1.42	Methyl Pentane	285
1.45	Benzene	20 ✓
1.60	n-Hexane	1000 (to ppb from only)
1.94	3-Hexanone	19
1.99	2-Hexanone	14
2.07	Toluene	26

\*Relative to Chloroform

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Table 14  
Llangollon Landfill Sample No. 7, Acid Extract  
Amoco PW 3 Well  
(Total Number of Peaks > 1 PPb 4)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
0.42	2-Ethylhexanoic acid (IS)	--
0.76	Unidentified	5
0.91	Methyl palmitate	1
1.0	Diethyl phthalate + Methyl Stearate	< 1
1.14	Bleed	--
1.21	Di(2-ethyl hexyl)phthalate	< 1
1.27	C <sub>15</sub> Terpeneol	< 1

\*Relative to Methyl Stearate

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Table 15

Llangollon Landfill Sample No. 7, Base Extract

Amoco PW 3 Well

(Total Number of Peaks &gt; 1 PPb 12)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
0.14	Diethoxyethane	15
0.33	Dichlorobutane	27
0.34	Identified	2
0.42	"	40
0.46	"	16
0.51	Camphor (IS) <sup>cal</sup>	--
0.74	Identified	7
0.76	"	2
1.0	Dibutylphthalate	1
1.11	Bleed	--
1.15	Bleed	--
1.21	di-(2-ethyl hexyl)phthalate	2

\*Relative to Methyl palmitate

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Table 16  
Llangollon Landfill Sample 8, VOA  
Artisan Water Well #2  
(Total Number of Peaks > 1 PPb 11)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>	<i>Chloro bottle</i>
0.44	Methylene Chloride	> 2500	✓ (not in methylene bottle)
0.68	Acetone	80	
0.87	Dichloroethane	1	
1.00	Chloroform	770	✓ (1/3 due to bottle)
1.24	Hexene-1	80	
1.41	Methyl pentane	600	
1.55	Diisopropyl ether	< 1	
1.59	n-Hexane	70	
1.95	Tetra chloroethene	< 1	
2.02	Toluene	< 1	

\*Relative to Chloroform

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Table 17  
Llangollen Landfill Sample No..8, Acid Extract  
Artisian Water Well #2  
(Total Number of Peaks > 1 PPb 1)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
0.42	2-Ethyl hexanoic acid (IS) <i>add</i>	--
0.92	Methyl palmitate	< 1
1.00	\ Dibutyl phthalate + Methyl Stearate	< 1
1.15	Bleed	--
1.22	q Di-(2-ethyl hexyl)phthalate	2
1.28	C <sub>15</sub> Terpeneol	< 1

\*Relative to Methyl Stearate

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Table 18  
Llangollon Sample No. 8, Base Extract  
Artisian Water Well #2  
(Total Number of Peaks > 1 PPb 3)

<u>Relative Retention Time*</u>	<u>Compound</u>	<u>Approximate Level (PPb)</u>
0.12	Diethoxyethane	4
0.33	Dichlorobutane	19
0.35	Unknown	1
0.51	Camphor (IS)	--
1.00	( Dibutyl phthalate	< 1
1.22	( di-(2-ethyl hexyl) phthalate	< 1

\*Relative to Methyl Stearate

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Table 19  
Overall Ratings of Organic Contaminants in  
Groundwater Samples from Llangollon Landfill

Sample	Location	Number of Peaks		1 ppb BASE	Overall Ratings*
		VOA	ACID		
No. 8	Artisian Well #2	11	1	3	A
No. 2	Monitoring Well #42	20	7	7	B
No. 7	Amoco Well PW3	24	4	12	C
No. 6	Recovery Well #3	28	5	9	D
No. 4	Recovery Well #27	27	22	40	E
No. 3	Recovery Well #29	29	41	42	F

\*A refers to the cleanest and F the most contaminated.

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